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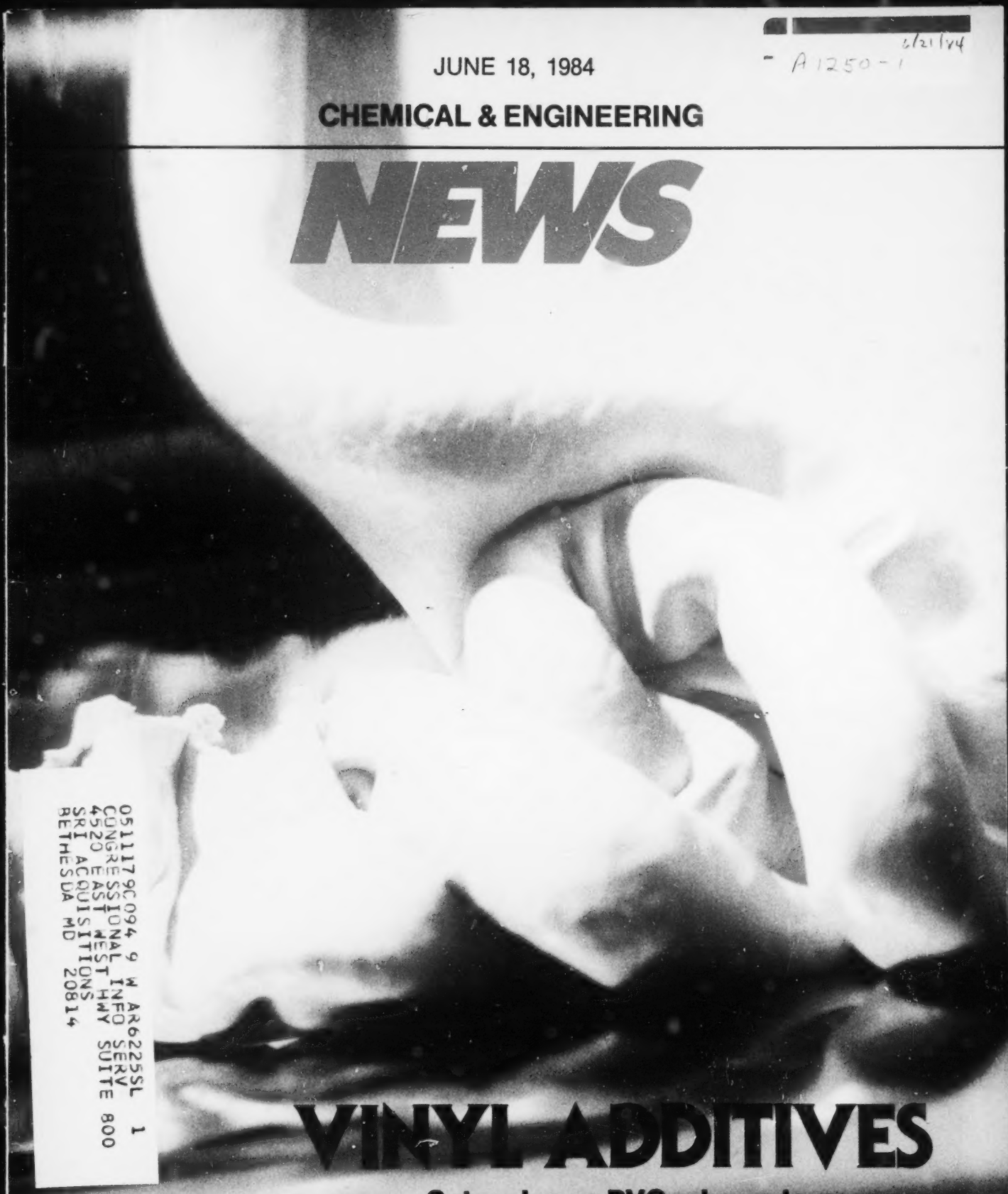
JUNE 18, 1984

CHEMICAL & ENGINEERING

NEWS



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VINYL ADDITIVES

Sales rise as PVC rebounds

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CHEMICAL & ENGINEERING NEWS

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Cover: Photo courtesy of Argus Chemical division, Witco Chemical

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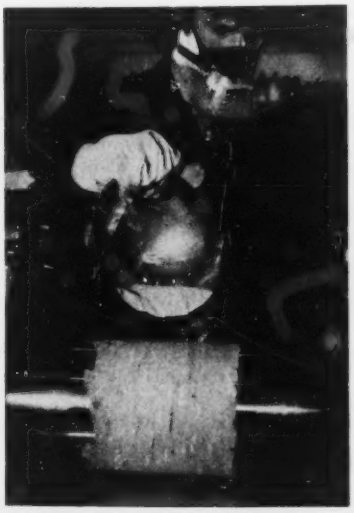


Plastics Additives Report

Additives are doing a good business, as sales for polyvinyl chloride increase and compounders use larger amounts of more additives. **Page 27**

House Acting Fast On Fiscal 1985 Money Bills

Well ahead of deadlines, the House has voted on budgets for all but two of the major research-funding agencies, rearranging some priorities

Janice R. Long, C&EN Washington

For the second year in a row the House is proving that when it wants to it can move very fast indeed. Even before the June 15 deadline for reporting appropriation bills, setting actual funding levels, had arrived, the House had passed a number of fiscal 1985 money bills. In fact by June 7 only the budgets for two of the major research funding agencies—the Department of Defense and the National Institutes of Health—had not been voted on.

As usual the House is setting its own budget priorities, adding some money here, taking some away there from the amounts requested by the Administration for the various agencies. Even when the totals come out to the original number, there's a better than even chance that some rearranging of budget priorities and funding has taken place. House Republicans, concerned that in many cases the totals are higher than what the Administration wanted to spend, have been offering floor amendments requiring across-the-board reductions in several appropriation bills.

The House passed its first fiscal 1985 appropriation bill, H.R. 5653, covering energy and water development in May. It contains almost $2 billion for the Department of Energy's energy supply R&D activities. (Fossil energy R&D is not included in that amount since its funding is set in the Interior appropriation bill.) That's a scant 1% less than DOE has available in this fiscal year and 2% less than the Administration requested. But the House did perform some major surgery on the budget request.

For instance, the Administration had requested a 3% increase in funding for magnetic fusion research; what the House approved was an 11% decrease to $419 million. In some areas, such as solar energy, electric energy systems, and storage and geothermal energy, the House approved a funding level higher than that requested by the Administration but still lower than this year's funding levels, repeat-

House pares most R&D agencies' budget requests

$ Millions	1985 Appropriation	1985 Request	1984	% change 1984–85
NASA	**$ 7,491.4**	**$ 7,491.4**	**$ 7,197.5**	**4.1%**
Space-flight control and data communication	3,602.8	3,600.3	3,791.6	−5.0
R&D	2,422.6	2,400.1	2,011.9	20.4
Research and program management	1,316.0	1,331.0	1,238.5	6.2
Facilities	150.0	160.0	155.5	−3.5
Energy	**3,762.2**	**3,974.0**	**3,479.7**	**8.1**
Energy supply R&D	**1,997.0**	**2,037.9**	**2,012.7**	**−0.8**
Nuclear	613.0	623.0	674.6	−9.1
Supporting research and technical analysis	493.8	479.6	395.1	25.0
Magnetic fusion	419.1	483.1	470.7	−11.0
Environment	226.2	226.2	216.0	4.7
Solar	174.5	163.6	181.6	−3.9
Electric energy systems and storage	40.3	35.3	44.2	−8.8
Geothermal	30.1	27.1	30.5	−1.3
Weapons R&D	**1,038.3**	**1,085.1**	**825.8**	**25.7**
General science and research	**726.9**	**851.0**	**641.2**	**13.4**
NSF	**1,499.8**	**1,501.8**	**1,320.3**	**13.6**
Research and related programs	1,293.2	1,308.2	1,140.3	13.4
Antarctic programs	115.1	115.1	102.1	12.7
Science education activities	88.7	75.7	75.0	18.3
Scientific activities overseas	2.8	2.8	2.9	−3.4
EPA	**1,301.3**	**1,198.7**	**1,111.5**	**17.1**
Salaries and expenses[a]	672.3	639.3	574.9	16.9
Abatement, control, and compliance	436.0	396.0	393.9	10.7
R&D	193.0	163.4	142.7	35.2
Commerce	**1,287.9**	**1,030.6**	**1,157.3**	**11.3**
NOAA	1,069.9	802.9	961.2	11.3
NBS	120.5	126.1	115.7	4.1
PTO	97.5	101.6	80.4	21.3
USDA	**745.8**	**761.5**	**733.8**	**1.6**
Agricultural Research Service	482.5	483.0	476.3	1.3
Cooperative State Research Service	251.8	266.8	247.6	1.7
National Agricultural Library	11.5	11.7	9.9	16.2
TOTAL	**$16,088.4**	**$15,958.1**	**$15,000.1**	**7.2%**

a Includes in-house research funding.

ing what has become a common pattern in recent years.

Conversely, the House added more money to the requested 21% increase for DOE's supporting research and technical analysis activities, which include chemical and materials research, to ensure the continuation of what have become controversial projects. The additions include $9.2 million for completion of the vitreous state laboratory at Columbia University; $3.0 million to continue the national center for chemical research at Catholic University; and $7 million for establishment of a supercomputer center at Florida State University. Funding for all other supporting research was set at the level requested by the Administration (C&EN, Feb. 13, page 6).

Next to come up for a floor vote was H.R. 5713, the Department of Housing & Urban Development-Independent Agencies appropriation bill. This bill, though it's hard to tell from the title, sets funding for the National Science Foundation, National Aeronautics & Space Administration, and Environmental Protection Agency.

The House-approved funding level for NSF is $1.5 billion, $2 million less than the budget request but $180 million more than is available this year. Funding for research and related activities is set at $1.3 billion, $15 million below the request but $153 million above this year's level. The House did a lot of rearranging within the research budget request, doubling the $20 million request for advanced scientific computing and the $2.5 million request for research improvement in minority institutions. It reinstated the experimental program to stimulate competitive research, funding it at a level of $1 million.

Inasmuch as the total provided for research activities is less than the amount requested by the Administration, something had to be cut to offset the above additions. That cut turned out to be $44 million from the budget requests for mathematical and physical sciences; engineering; astronomical, atmospheric, Earth, and ocean sciences; and program development and management areas to be allocated at NSF's discretion.

The bill also includes $88.7 million for the foundation's science education activities, $13 million more than was requested to hold spending essentially constant in fiscal 1985. The House additions are $8 million to increase both the number and annual stipend of graduate fellowship grants, which were already slated for an increase, and $5 million for instructional instrumentation for four-year colleges.

NASA's total budget, as approved by the House, is $7.5 billion, an increase of only 4% over fiscal 1984. However, the R&D budget would be up 20%, or $411 million, to $2.4 billion, $22.5 million more than requested. Included in the bill is the $150 million requested for development of a permanently manned space station.

The House added to the budget request $40 million for flight demonstration of the advanced communication technology satellite, $10 million each for physics and astronomy and planetary research and analysis; continued work on the advanced turboprop programs; and advanced work on a "shuttle derived" expendable launch vehicle. It subtracted $10 million from the aeronautics research and technology base request and told NASA to find some way to save another $37.5 million.

Under the bill, funding for EPA extramural R&D programs is set at $193 million, 18% higher than the request and 35% above fiscal 1984 funding levels. The $30 million addition to the budget request includes $5 million for health effects, exposure assessment, and biotechnology research; $5 million for exploratory research grants and centers; $4 million for hazardous waste control technology; $3.5 million for indoor air pollution research; and $1.5 million for acid rain research on atmospheric chemical transformation and transport processes.

These first appropriation bills passed with very little debate, but by the time the Commerce Department's appropriation bill, H.R. 5712, hit the floor the budget cutters were ready. They succeeded in getting an amendment approved that called for an across-the-board reduction of 4% for each line item of funding in the bill. That still left enough money for an increase of 11% in the National Oceanic & Atmospheric Administration budget, which the Administration wanted to cut 16%. Among the $312 million worth of additions to the budget request were $35 million for the Sea Grant program, $3.8 million for the Great Lakes environmental research laboratory, and $800,000 for acid rain research.

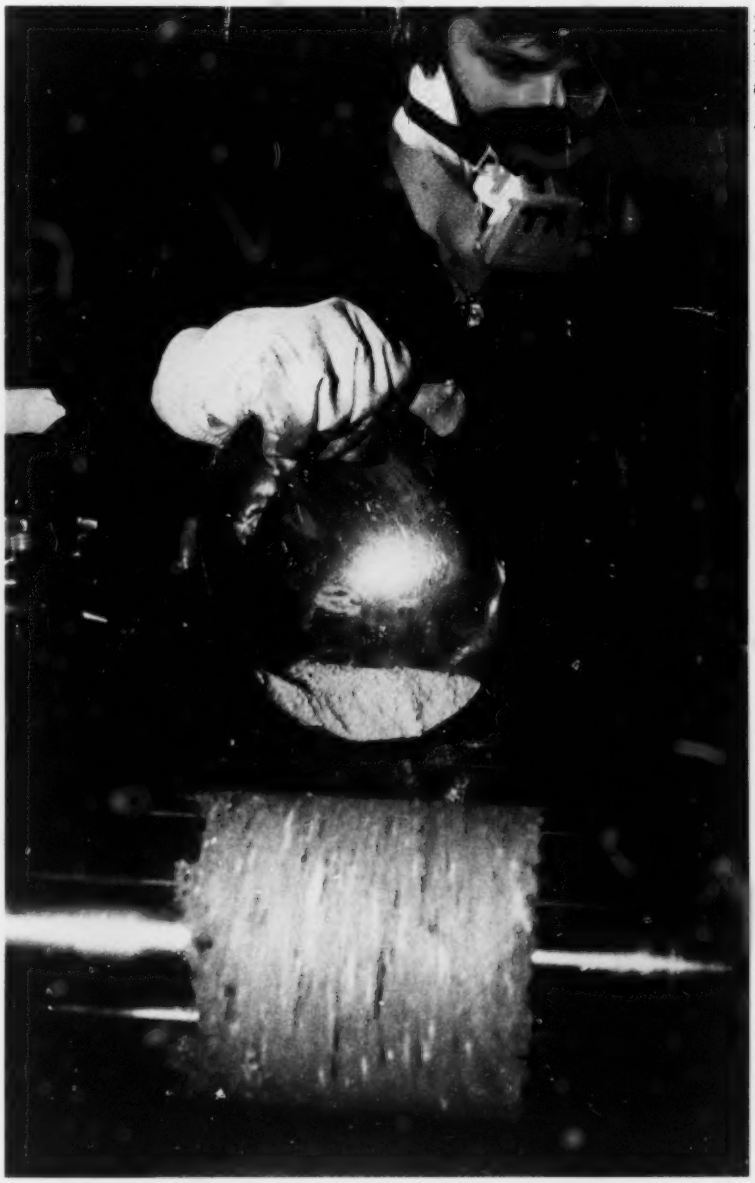
The reduction only made things worse, however, for the National Bureau of Standards. The Administration had asked for a 9% funding increase. The Appropriations Committee had approved a 10% increase. But the bill, as approved by the full House, allows only a 4% increase to $121 million. The bill does restore funding for the Center for Building Technology, the Institute for Computer Science & Technology, and the Center for Fire Research. However, funding for the Patent & Trademark Office would increase 21% to $97.5 million, buttressed by fees collected for processing patent and trademark applications.

The agriculture appropriation bill, H.R. 5743, also was the subject of an across-the-board cut, but of only 1%. The House set funding for the Agricultural Research Service at $483 million, $1 million over the request and $6 million over this year's level. It earmarked specific amounts for such things as feeds, hornfly, guar, bee, cherry dieback, and sugarcane research. It also endorsed spending $200,000 for a continued assessment of dioxin contamination in the Great Lakes food chain.

The House decreased the budget request for the Cooperative State Research Service by $15 million to $252 million. It reserved $17.1 million for special research grants in 40 research areas and in many cases stated where the money was to be spent. And, as usual, it cut the budget request for competitive research grants by $17 million to $32.2 million.

The House does not have the final, only the first, say in setting agency budgets. The Senate is still to be heard from, but that body is usually at least as generous, if not more so, as the House when it comes to funding research programs. □

Additives Sales Buoyed By PVC Recovery


Argus Chemical photo

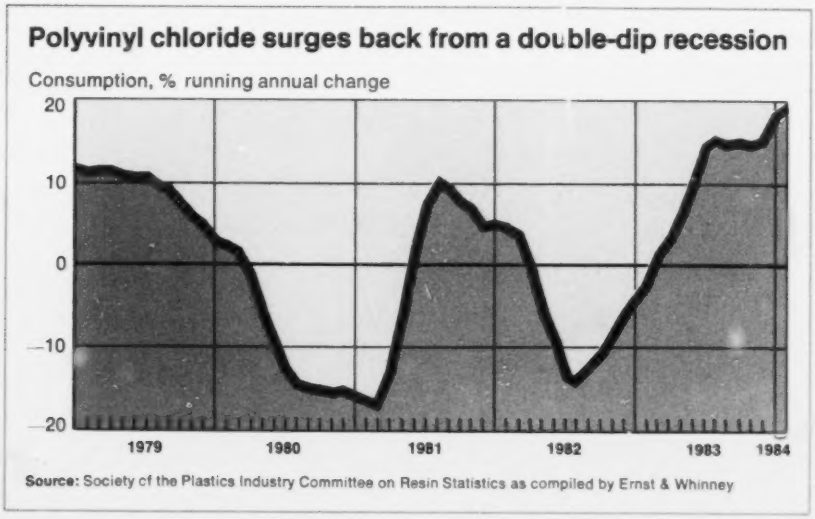
Argus Chemical technician prepares PVC compound for evaluation

Stephen C. Stinson, C&EN New York

Sales of polyvinyl chloride in the U.S. are charging back from a double-dip recession that began in 1979, buoying up with them the fortunes of companies that sell chemical additives to compound PVC. U.S. sales of PVC resin could jump 15% in 1984 to hit a record 6.3 billion lb. Because larger amounts of more additives are put into PVC by greater numbers of compounders than for any other resin, stakes are high on the health of this one plastic.

By itself, PVC is a hard, brittle resin that is almost useless, and its thermal instability makes it difficult or impossible to process. That PVC has become a high-volume plastic capable of assuming limitless useful forms is a triumph of chemical innovation. Elliott Weinberg of Cross Gates Consultants, East Brunswick, N.J., points out that properties of PVC are uniquely susceptible to additives, which render it anywhere from rigid and resilient to flexible and supplely draped, and make it useful for applications ranging from a low-cost plastic for novelties to an engineering resin. Because minute changes in additive types and amounts drastically alter processibility and end-use properties, many PVC producers have set up compounding operations, a large number of custom compounders has sprung up, and every processor who does not already compound its own resin ponders the advisability of doing so.

Weinberg, who is also a principal in the PVC technology transfer organization Technical Information Exchange of East Brunswick, says that PVC is an ideal beginning for plastics industries in developing countries. This is because such countries can progress from importing



compounds to domestic compounding to resin production. Also, low-cost, low-energy processing techniques are available. A $50,000 extrusion line can make pipe or hose. Liquid dispersions of PVC in plasticizer can be poured into molds for footwear or coated onto fabrics, which are then cured in ovens. And though film calendering lines often cost $5 million to $7 million, film also can be blown from an extruder.

PVC is a success story that might never have happened. The plastic has been beset by problems since its beginning, and new problems have cropped up to the present day. Each problem has been met by a chemical solution, and chemistry will be crucial to meet current concerns over smoke suppression during burning and alleged carcinogenicity of some plasticizers.

Weinberg points out that whereas other resins developed in an orderly manner with a firm underpinning of science, PVC started as an afterthought and grew haphazardly from there. Today there are heat stabilizers that allow melt processing of PVC and use of products at elevated temperatures. But why PVC is unstable to heat, let alone why heat stabilizers work, is the subject of vigorous debate. And although about 100 plasticizers are commercially available, the theory of plasticization is too primitive to predict the improved ones the industry seeks.

PVC was developed by the German company Griesheim-Electron around 1912 in efforts to find uses for then-abundant acetylene illuminating gas. Chemist Fritz Klatte produced vinyl chloride by addition of hydrogen chloride and discovered its polymerization by organic peroxides two years later. Attempts to replace celluloid with PVC foundered over brittleness and heat instability, and in 1926, Griesheim-Electron even stopped paying the fees that maintained its patents.

Rediscovery of PVC happened that same year in the U.S., when Waldo L. Semon of B. F. Goodrich found it could be made flexible by boiling in tricresyl phosphate or dibutyl phthalate. Semon also hit upon white lead as a heat stabilizer, which continues in wide use today. At about the same time, chemists at Union Carbide were trying to find uses for the ethylene dichloride byproduct of ethylene chlorohydrin manufacture. They made vinyl chloride by treatment with sodium hydroxide.

In 1936, Union Carbide started producing vinyl chloride at the rate of 4 million lb per year, most of which went to Goodrich. Also in the mid-1930s, T. L. Gresham of Goodrich tried about 1000 compounds in search of better plasticizers and came up with di-2-ethylhexyl phthalate, which is still the workhorse today.

The long search for chemical ways to enhance workability and properties of PVC continues. For example, current theories of thermal instability propose that there are defect sites inserted into the PVC chain during polymerization that are vulnerable to dehydrochlorination on heating.

In one view of how defect sites arise, disproportionation of radicals could introduce carbon-carbon double bonds that would render chlorine atoms adjacent to them allylic. Heat-provoked eliminations of these chlorine atoms could begin a zipper mechanism, producing more double bonds and new allylic chlorines. The exothermic reaction and hydrogen chloride evolution could then blow the head off an extruder. If unstabilized PVC were extruded safely (which it can be by careful processing on multiscrew machines), further heating during use could continue dehydrochlorination, causing blackening owing to the conjugated polyene sequences generated.

William H. Starnes Jr., a polymer chemist at AT&T Bell Laboratories, Murray Hill, N.J., recently confirmed existence of a long-sought second candidate defect site (C&EN, April 30, page 25). His work suggests that a radical rearrangement forms a side chain on the growing polymer with a tertiary chlorine at the branch point. Tertiary chlorines also might be susceptible to dehydrochlorination and initiation of the zipper cascade.

The controversy continues, with some Soviet scientists suggesting that oxygen present during polymerization could form γ-chlorocrotonyl-type groups in the chain. Such activated γ-chlorines are the real starting points for degradation, they say.

Whatever the nature of the defect site, heat stabilizers seem to act by neutralizing them before they can spark the degradation reaction. Heat stabilizers currently used include lead phosphite, sulfate, or phthalate; mixed barium-cadmium or calcium-zinc soaps or phenoxides; mixed mono- and dialkyltin carboxylates or mercaptides; and antimony mercaptides. During processing, these compounds replace reactive chlorines with less reactive carboxylate, sulfate, phenoxide, or mercaptide groups. Cationic portions take up chloride ions. Stabilizer makers must use care in designing such compounds, because some metal

chlorides catalyze dehydrochlorination.

One chloride that does not is lead chloride. The associated nature of lead chloride also imparts good electrical resistance to PVC, leading to wide use of lead stabilizers in wire insulation and cable jacketing, according to William Daly, technical manager for stabilizers at American Cyanamid's Chicago location. Makers of cable jacketing use lead phosphite, he says, because it lends stability to ultraviolet light as well as heat stability to such outdoor applications. Inexpensive lead sulfate goes into wire insulation for service below 90 °C. For use temperatures to 105 °C, lead phthalate is more compatible with the less volatile polyester and trioctyl trimellitate plasticizers used in such insulation. Daly says there is increasing interest in coprecipitated lead sulfate-phthalate, because it could be used in all wire insulation, decreasing the need to keep inventories and install bulk handling systems for several stabilizers.

One outlet that would be a good market for lead stabilizers is PVC pipe. Cost-cutting pressures in this highly competitive field are intense, and lead stabilizers might be more cost-effective. The stumbling block is concern about leaching of lead from pipe used to carry drinking water. Approval of additives for such pipe would have to come from the National Sanitation Foundation. The foundation, based in Ann Arbor, Mich., is a private group formed to police the industry to avoid need for government regulations.

Several lead stabilizer makers have joined under the umbrella of the Lead Industry Association to submit data to the sanitation foundation showing that their products are not leached from pipe in dangerous amounts. Some observers tell C&EN that even if the foundation approves some lead stabilizers, pipe extruders may refrain from using them anyway, because of fears of public reaction. But an approval might still expand use of lead stabilizers for drain, waste, vent, and other pipe that does not carry drinking water. This is because producers of such pipe often also extrude foundation-approved pipe in the same plant and fear contamination of drinking water pipe from another part of the building. Approval of lead stabilizers for drinking water pipe might encourage pipe extruders to use them on their other lines.

What pipe producers do use today as stabilizers are antimony and organotin mercaptides. These products have undergone a tortuous evolution toward ever cheaper, more potent compounds and lower use levels as stabilizer producers have vied for the business of pipe producers. Just as PVC makes or unmakes the fortunes of the plastics chemical additives industry, so pipe of all kinds, which takes 45% of all PVC used in the U.S., rivets the attention of stabilizer producers. American pipe extruders likely will process 2.8 billion lb of PVC this year, using 15 million lb of stabilizers to do so.

The most recently developed type of low-cost pipe compound heat stabilizer is a mixture of the antimony trimercaptide of 2-ethylhexyl thioglycolate with calcium stearate. Known in the industry under the misnomer "antimony isooctylthioglycolate," this product was invented by Weinberg of Cross Gates Consultants when he was at M&T Chemicals, Rahway, N.J., and was further improved by Synthetic Products Co., Cleveland.

The impetus to develop the product, as it has been to improve the tin mercaptides, was the steadily escalating price of tin. The New York tin price rose from $1.67 per lb in 1971 to a peak of $7.73 in 1980, and was still $6.02 in 1983. By contrast, the price of antimony also has fluctuated over the years, but was only 90 cents per lb in 1983. Based on the number of available mercaptide groups in comparison with the also misnamed "dibutyltin isooctylthioglycolate," the antimony content costs only $36.50 per lb-equivalent, versus $357 for tin.

Although a few pipe extruders do use the antimony compound, some suppliers of this stabilizer admit that it has never become wide-

Of PVC consumption, 45% goes for pipe alone

Consumption, millions of lb[a]	
Construction	**3775**
Flooring	350
Panels & siding	305
Pool & pond liners	40
Roofing	30
Pipe & pipe fittings	2805
Windows & other profiles	175
Weatherstripping	45
Other	25
Packaging	**650**
Sheet	125
Film	310
Bottles	175
Closures & other uses	40
Electrical	**475**
Wire & cable	400
Connectors & plugs	60
Other	15
Furnishings	**365**
Upholstery	105
Wall covering	50
Appliances	105
Window shades, blinds & awnings	35
Tablecloths & mats	50
Other	20

Consumption, millions of lb[a]	
Transportation	**235**
Upholstery, trim & tops	135
Floor mats	25
Bumper parts	25
Other	50
Recreation	**195**
Records	105
Toys	45
Other	45
Apparel	**185**
Footwear	95
Handbags	30
Luggage	35
Other	25
Medical	**150**
Tubing	50
Blood & intravenous bags	50
Other	50
Miscellaneous	**290**
Credit cards	25
Garden hose	50
Traffic cones	20
Tool handles	15
Adhesives & sealants	90
Other	90
TOTAL	**6320**

[a] U.S. consumption, 1984.

Heat stabilizers offer trade-off of properties

Suitability rating[a]	Lead	Barium-cadmium soaps	Barium-cadmium liquids	Barium-cadmium-zinc liquids	Calcium-zinc	Dialkyltin carboxylates	Dialkyltin mercaptides
Process stability	3	2	2	2	1	1	3
Process color stability	1	2	2	3	1	1	3
Moderate-temperature color stability	1	3	2	3	1	2	3
Moderate-temperature properties retention	3	2	1	1	1	2	3
Electrical properties	3	2	1	1	0	0	1
Clarity	0	1	3	2	3	3	3
Light stability	3	3	3	3	2	3	0
Toxicity	0	0	0	0	3	3	2
Odor	3	3	1	1	3	2	0
Sulfide stain	0	1	1	2	3	3	3
Cross-stain	0	1	1	2	3	3	0

[a] 3 = best available, 2 = generally suitable, 1 = marginal, 0 = unsuitable. **Source:** Cdlorite Plastics Co.

spread. This is because it requires reformulation, initially is difficult to get good results with, yields lower pipe output rates, and is less forgiving compared with tin.

The tin stabilizers used in pipe extrusion and processing of rigid PVC generally are based on mixtures of methyltin and dimethyltin or butyltin and dibutyltin. Octyltin-dioctyltin compounds are approved for food packaging and medical applications because they are less extractable. Mercaptide portions have been 2-ethylhexyl thioglycolate, though such so-called reverse esters as β-mercaptoethyl oleate have been found cheaper and more potent, though more odorous, for pipe.

Alkyltin compounds enhance early color retention, whereas dialkyltins promote long-term stability, explains Victor Struber, marketing manager of Argus Chemical division of Witco Chemical. This may be because alkyltins have more mercaptide per mole to cap defect sites than dialkyltins, though the product alkyltin trichloride is a stronger Lewis acid than dialkyltin dichloride and thus would catalyze later blackening if used alone.

Another cost-cutting move has been to lower tin content by using greater than stoichiometric amounts of mercaptoester. In such cases the tin functions as a catalytic carrier. But Argus' Struber cautions that there is a certain irreducible amount of tin needed for effectiveness, perhaps 8%. Stabilizer producers have tried for years to develop all-organic stabilizers with no metal. But hope for success fades as chemists fear that costs of organic synthesis could equal or exceed those of metal.

For flexible PVC, barium-cadmium compounds, with or without zinc, are favored heat stabilizers. Calcium-zinc types go for food packaging or medical applications, though they are less effective. The reason for using these stabilizer types is that they are lower in cost than tins, explains Donald G. Hampson, general manager for thermoplastic additives at the Interstab Chemicals subsidiary of Akzo Chemie America. They do not process so well as tins in rigid compounds, however, so they have not been used much for rigid PVC in the U.S. The plasticizer content of flexible compounds makes them more easily processible, so compounders of these can take advantage of lower costs of barium, cadmium, calcium, and zinc.

Analogous to the tins, cadmium and zinc are good for early color retention, though their chlorides are strong Lewis acids. Barium and calcium contribute long-term stability. Phosphites like tris(nonylphenyl) phosphite and epoxy plasticizers like epoxidized soybean, linseed, and tall oils are synergistic with metal soaps and phenoxides. Stabilizer makers often supply a metal-phosphite blend, and there have been attempts to market metal-phosphite-epoxy combinations. But Struber of Argus points out that compounders' needs are so individual that one ratio of the three would be too limiting.

A recent research push has occurred for stabilizers in PVC blood- and intravenous-solution bags and medical tubing that withstands sterilization by γ-rays. This is because radiation may replace ethylene oxide for sterilization of medical goods. The high flux of γ-rays could initiate radical reactions in PVC that would degrade the resin in ways similar to heat. Most stabilizer makers are reticent to reveal their formulations, but they must be based on such Food & Drug Administration-approved ingredients as calcium, zinc, epoxidized soybean oil, and dioctyltin isooctylthioglycolate. Robert G. Weiler, who is Interstab's business development manager for rigid PVC, says that Interstab gets good results by increasing the content of zinc, epoxidized soybean oil, and antioxidant.

Some of the same compounds that are effective heat stabilizers also function as "kickers" for chemical blowing agents. Azodicarbonamide is the agent most used in PVC. Zinc compounds, especially zinc oxide, are good accelerants. The most potent are compounds of lead, though compounders tend not to use these out of concern for possible toxicity. Rhomie Heck, technical service specialist in plastics blowing agents at Uniroyal Chemical, says that 10% of zinc oxide in azodicarbonamide lowers the blowing temperature to 175 to 180 °C from 210 °C. Blowing agents usually are used with plastisols, which are liquid dispersions of PVC in plasticizer. For these, it is important that the blowing temperature of the blowing agent match the curing temperature of the plastisol. Plastisol processors generally work at temperatures from 150 to 200 °C.

In addition to stability to heat, outdoor applications of PVC sometimes require UV absorber additives to protect against sunlight. In pigmented, pastel applications like some vinyl siding, window frames, and building panels, a 15 to 20% loading with titanium dioxide is protection enough. For clear, translucent, or darkly pigmented PVC, high loadings of titanium dioxide are ruled out, and the UV absorbers

A history of polyvinyl chloride

1835. Justus von Liebig at the University of Giessen discovers that reaction of the "oil of the Dutch chemists" (ethylene dichloride) with alcoholic potash produces a chlorine-containing gas. Liebig assigns confirmation of the work to his student, Victor Regnault, and lets Regnault publish as sole author. As a result, Liebig is forgotten as the discoverer of vinyl chloride.

1872. E. Baumann finds that exposure of vinyl chloride to sunlight in a sealed tube produces a white solid that resists attack by potassium hydroxide or water and melts with decomposition above 130 °C.

1912. Chemische Fabrik Griesheim-Electron gives chemist Fritz Klatte the task of finding uses for acetylene, and he develops commercial synthesis of vinyl chloride by addition of hydrogen chloride.

1915. Klatte finds that organic peroxides initiate polymerization to polyvinyl chloride.

1926. After years of effort to develop rigid PVC as a replacement for celluloid, German workers have failed to build machinery that will process the resin and have not overcome the problem of thermal instability. Griesheim-Electron stops paying the fees that maintain its patents. Meanwhile in the U.S., Union Carbide, looking for uses for the ethylene dichloride by-product of chlorohydrin production, converts it to vinyl chloride with sodium hydroxide. PVC also interests Waldo L. Semon at B. F. Goodrich, who is looking for an adhesive to bond rubber to metal. Semon observes that boiling PVC in such liquids as tricresyl phosphate or dibutyl phthalate renders it highly elastic, thus discovering plasticizers. In addition to molding parts from plasticized PVC, Semon goes on to coat wire, cloth, and metal parts with solutions of PVC in plasticizer or plasticized PVC in organic solvents, thereby inventing plastisol and organosol processing techniques. He uses white lead and sodium silicate as heat stabilizers.

1928. Union Carbide and Du Pont each find that copolymers of vinyl chloride with vinyl acetate have improved processibility.

1934. Frazier Groff of Union Carbide discovers that alkaline earth soaps stabilize PVC to heat.

1936. Union Carbide's PVC homopolymer plant comes on stream at 4 million lb per year. Chemists there find that dialkyltin soaps are also heat stabilizers. At B. F. Goodrich, T. L. Gresham tests thousands of compounds before settling on di-2-ethylhexyl phthalate as the best plasticizer.

1941. During World War II, PVC is commandeered for military use, with the Navy in particular using large amounts of phosphate-plasticized, fire-retardant wire insulation developed by General Electric.

1947. One or more workers now unknown hit upon synergistic combinations of barium, cadmium, calcium, and zinc soaps as heat stabilizers.

1952. Elliott Weinberg at Metal & Thermit Corp. invents the dibutyltin dimercaptide with 2-ethylhexyl thioglycolate as a heat stabilizer for rigid PVC, and at the same time invents the antimony trimercaptide low-cost stabilizer for pipe, which will be commercialized 30 years later as tin prices skyrocket.

1960. U.S. production of PVC tops 1 billion lb.

1970. Pier Luigi Viola at Regina Elena Institute for Cancer Research in Rome finds that exposure to very high levels of vinyl chloride vapor causes ear tumors in rats. U.S. production of PVC tops 4 billion lb.

1973. Cesare Maltoni, working for a group of European firms at the Institute of Oncology in Bologna, Italy, finds that inhalation of vinyl chloride vapors causes rare angiosarcoma liver cancer in rats. The Manufacturing Chemists Association is allowed to see his work, but only under a confidentiality agreement. Forbidden to communicate Maltoni's findings, MCA commissions its own studies of rats and worker exposures. Late in the year, physician John L. Creech links deaths of two B. F. Goodrich workers from angiosarcoma to vinyl chloride exposure in Louisville, Ky. Eventually, angiosarcoma deaths of three dozen people worldwide are tied to vinyl chloride exposure.

1974. Goodrich tells the National Institute of Occupational Safety & Health of the cancer link. The Occupational Safety & Health Administration reduces permissible vinyl chloride exposure to 50 ppm from 500 ppm and proposes a "zero detectable limit." Industry spokesmen say that will force them to shut down. OSHA agrees to a phased reduction to 1 ppm. PVC makers announce that they can meet this and alter their plants to do so. Later, activists tar industry for saying first that the OSHA limit would shut them down and then agreeing that they can meet it.

1980. The National Cancer Institute finds evidence that di-2-ethylhexyl phthalate plasticizer causes cancer in rats and mice.

1982. Stung by inroads of PVC into pipe, metal-pipe makers counterattack with a campaign to warn public authorities of toxicity of gases emitted by PVC in fires.

1984. A recovering U.S. economy carries PVC use to a record 6.3 billion lb.

most used then are benzophenones and benzotriazoles. Also used are such acrylates as 2-ethylhexyl 2-cyano-3,3-diphenylacrylate, which are also called acrylonitriles.

According to Joseph Stretanski, who is technical manager for plastics additives for American Cyanamid, benzophenones such as 2-hydroxy-4-methoxy- or 2-hydroxy-4-octoxybenzophenone usually are used in conjunction with barium-cadmium stabilizers, and such benzotriazoles as 2-(2-hydroxy-5-*tert*-octylphenyl)benzotriazole are used with tins. Benzophenones are yellow and contribute color to a clear vinyl compound. In applications such as a clear vinyl coating for a richly colored flooring tile, the slight yellowing may not matter. Benzotriazoles perform better than benzophenones and yield clear vinyl products that are water-white. So some

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processors prefer benzotriazoles, despite their higher cost, than many benzophenones.

Despite the great attention given to developing chemical additives to stabilize PVC in processing and in use, plasticizers are the additives that are used in greatest amounts for this resin. In recent years, applications for rigid PVC have grown faster than for flexible PVC, as in the invasion of the pipe market. Still, as Edward J. Wickson reminded the CAN-AM Chemical Congress earlier this month in Montreal, more PVC is processed on a flexible compound basis than is rigid. Wickson is chief products applications scientist at Exxon Chemical's intermediates technology division in Baton Rouge. He told his Montreal listeners that in 1982, the U.S. used 3.0 billion lb of plasticized compounds, containing 2 billion lb of PVC and 1 billion lb of plasticizers, compared with 2.6 billion lb of rigid compounds.

Since the emergence of di-2-ethylhexyl phthalate ("dioctyl phthalate," or DOP) as plasticizer of choice 50 years ago, researchers have tried to find new ones to replace it. Wickson used figures of the U.S. International Trade Commission to document that they have been partly successful, as DOP declined to 25% of total U.S. plasticizer use in 1982, compared with 42% in 1972. New plasticizers have been found, usually costing more than DOP, but superior to it in extractability, volatility, low-temperature flexibility, or fire retardance. Still, Wickson made a case that his firm's diisononyl phthalate is slightly more cost-effective than DOP.

Plasticizers are liquids that are absorbed by PVC particles during compounding to produce dry, free-flowing powders or pellets even at high use levels, when there may be much more plasticizer than resin. Processing these compounds produces a close physical association among plasticizer molecules and PVC chains to yield flexible films and objects from which a good plasticizer does not exude. Even a "worm" made as a fishing lure from highly plasticized PVC is dry, despite its realistic feel. Although plastisols are processed as liquid, dispersions of PVC microparticles in plasticizer cure to dry parts on oven curing.

Plasticization and the efficacy of plasticizers are as much mysteries as the heat instability of PVC and the workings of heat stabilizers. Vincent P. Kuceski has described the lubricity, gel, and free-volume theories of plasticization in American Chemical Society Short Courses. Kuceski, who is vice president of research and development at C. P. Hall Co., Chicago, says that plasticization in the lubricity view is the dissociation of crystalline regions of polymer by plasticizer molecules. To be efficient at this,

Plasticizer choice depends on properties desired

Relative performance rating[a]	General-purpose phthalates	High-molecular-weight phthalates	Epoxidized soybean oil	Trioctyl trimellitate	Polyesters
Exudation[b]	5	3	2	4	1
Humidity resistance	5	3	2	4	1
Acid resistance	5	4	2	3	1
Dry-blend mixing rate	5	3	2	4	1
Melt-mixing fusion rate	5	2	1	4	3
Plastisol viscosity	5	3	2	4	1
Volatility[b]	1	2	4	5	3
Light stability[b]	5	3	1	4	2
Migration resistance[b]	1	2	4	3	5
Extraction resistance (kerosine)[b]	2	1	4	3	5
Fungus resistance[b]	4	3	2	5	1
TOTAL RATING	**43**	**29**	**26**	**43**	**24**
PERMANENCE RATING TOTAL	**18**	**14**	**17**	**24**	**17**

a 5 = best, 1 = poorest. b Measure of permanence. **Source:** Nuodex

the plasticizer must be compatible with the resin overall, but not too good a solvent. This is because plasticizer molecules serve as physical "spacers" among PVC chains.

In the gel theory, PVC stiffness arises from points of intermolecular attraction among chains, caused perhaps by association of polar carbon-chlorine bonds, or from chain entanglements. Plasticizer molecules might then neutralize bond polarity or intercalate among chains to break up entanglements. To do this, the plasticizer must be a fairly good solvent for PVC to prevent the plastic from "precipitating out."

In the free-volume theory, thermal motions of small, mobile plasticizer molecules increase the amount of volume in the matrix by an amount greater than they themselves occupy at a given instant. PVC chains are free to use this volume to increase their mobility. But plasticizers can do this only if they are not very good solvents for PVC, because plasticizer molecules must be free to move and increase free volume rather than be constrained by interaction with PVC chain sites.

Despite progress in understanding plasticization, Kuceski says that chemists are far from being able to calculate how much of a given plasticizer to use to lower the glass transition temperature of PVC by a desired amount or to predict what compounds would be good plasticizers. He suggests that it may be possible to develop a computer program to do this that would take into account solubility parameters, free volume, and donor-acceptor values of species involved in dipole-dipole interactions.

Plasticizers are classified as primary or secondary. Primary plasticizers are very compatible with PVC and can be used as sole plasticizers. These tend to be aromatic, aliphatic, epoxidized, or phosphate esters. Secondary plasticizers have low efficiency or compatibility with PVC and only supplement primary ones. Examples are hydrocarbon oils, added to reduce costs, or chlorinated paraffins for fire retardancy.

As criteria for plasticizer selection, Kuceski cites compatibility, permanence, performance at low temperatures, efficiency, processibility, esthetic and physical properties, durability and stability, and cost. He defines compatibility as ability to form a homogeneous mixture with PVC that does not exude plasticizer from finished product. Compatibility is enhanced for branched-chain, compact, or low-molecular-weight molecules, and by presence of ether or polyester groups. It is greater at high temperatures for monomeric plasticizers and at low temperatures for polyesters.

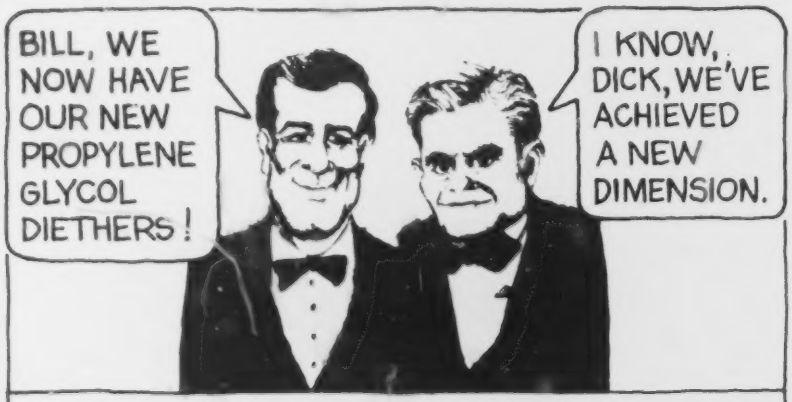
To Kuceski, permanence includes resistance to physical changes by heat, humidity, solvents, oils, and weather that might volatilize or extract plasticizer or cause migration to another polymer. Others in the plasticizer industry include chemical changes by heat, weather and light, salt water, chemical attack, fungus, and bacteria. Kuceski uses the term durability and stability to describe resistance to such chemi-

Continued on page 47

Prices of plasticizers vary widely

1982 price, $ per lb	
Aromatic	
Di-2-ethylhexyl phthalate	$0.40
Diisodecyl phthalate	0.42
Di-*n*-octyl phthalate	0.37
Ditridecyl phthalate	0.63
Tri-2-ethylhexyl trimellitate	0.65
Aliphatic	
Di-2-ethylhexyl adipate	0.61
Di-2-ethylhexyl sebacate	1.50
Polymeric	0.96
Epoxy	
Epoxidized linseed oil	0.83
Epoxidized soya oil	0.48
Fire retardant	
Chlorinated paraffin, 35 to 64% chlorine	0.37
65% chlorine and up	0.52
Phosphate esters	0.50
Polyvinyl chloride[a]	0.24

[a] Included as reference. **Source:** U.S. International Trade Commission



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Efficiency is the amount of plasticizer that must be used to get a desired value of properties such as glass transition temperature or a 1500-psi modulus at 100% elongation. Low-molecular-weight, aliphatic plasticizers enhance efficiency.

As for durability and stability, branched chains are less susceptible to bacterial or fungal attack than are linear esters. Fatty acid derivatives used as lubricants in PVC compounds also are subject to attack. For PVC film and parts to be used in humid environments, compounders add a biocide. The one most used is 10,10′-oxydiphenoxarsine, though there is some use of trialkyltin compounds too. Plasticizer oxidation also can occur at high processing temperatures, and Kuceski says that his firm sometimes adds antioxidants to plasticizers.

Though FDA approval is unnecessary for all applications, there are plasticizers that have wide approval for food packaging and medical uses. These include di-2-ethylhexyl phthalate, adipate, and sebacate, many polyesters, and epoxidized oils. There has been concern over possible carcinogenicity of di-2-ethylhexyl phthalate and adipate. Speaking in Montreal, Exxon Chemical's Wickson reviewed findings of the National Cancer Institute that these compounds are carcinogenic in rats and mice. But Wickson also pointed out that work at ICI suggests that di-2-ethylhexyl phthalate metabolism is different in primates. The Chemical Manufacturers Association has commissioned a study of plasticizer toxicity and carcinogenicity. FDA has not yet changed the regulatory status of 2-ethylhexyl esters, but some processors have begun evaluating other phthalates and adipates as a precaution.

Di-2-ethylhexyl phthalate has con-

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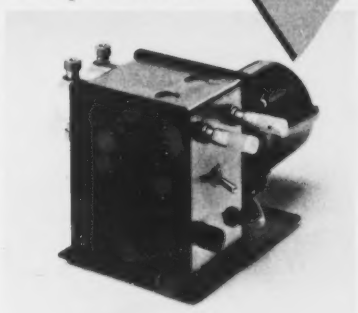

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tinued in wide use over the years because it is still the low-cost plasticizer. Nevertheless, as Wickson says, PVC has moved into high-performance markets that require better properties than general-purpose phthalates can give. These uses include single-ply roofing membranes, water bed heaters, automotive crash pads, refrigerator gaskets, electrical tapes, and oil-resistant cable. In addition, Underwriters Laboratories will issue new standards on residential wire insulation late this year, raising ratings to 90 °C from 60 °C and require heat-aging tests at 136 °C rather than at 100 °C as at present.

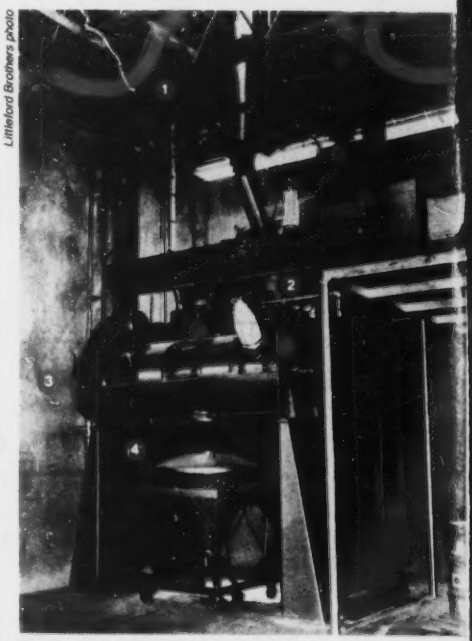
But as plasticizers impart flexibility to PVC, they also raise its flammability. The high chlorine content of rigid PVC makes it fire resistant enough not to need fire retardant additives. Compounding large quantities of plasticizer into the resin decreases the average chlorine content, however. Fire retardants most often used in flexible PVC are antimony oxide and phosphate ester and chlorinated paraffin plasticizers.

Antimony oxide is synergistic with the chlorine content of PVC, explains Philip Rakita, who is marketing manager at M&T Chemicals in Rahway, N.J. Antimony trichloride is thought to be formed as the plastic is heated. Vapors of this compound may stop the free-radical reactions of combustion. The oxide itself is a white pigment, however, and cannot be used in applications intended to be clear or pigmented in dark colors. M&T and other firms have gotten around this by offering sodium antimonate, which is not a pigment; antimony oxide of larger particle size, which does not have as much hiding power; and antimony pentoxide, which as a colloid also is not a pigment.

Chlorinated paraffins and such phosphates as tricresyl phosphate also make no contribution to compound color. Both types of fire-retarding plasticizer are used to replace only part of the plasticizer usually used, because of the poor compatibility of chlorinated hydrocarbons and the relatively high price and low efficiency of phosphates. Chlorine in chlorinated paraffins is synergistic with any antimony oxide used, but M&T's Rakita says that phosphate esters and antimony impede one another's fire-retarding action.

Having chosen the types and amounts to be used, the compounder next blends them with resin on an appropriate machinery line. Both rigid and flexible PVC compounds end up as solids, either pellets or powder, to be sent to such fabricating equipment as injection molding machines, extruders, or calenders. To produce a plastisol, the compounder uses a dispersion-grade PVC whose particle sizes are 20 μm or less. The result of mixing with

Dry blending yields low-cost powdered compounds



Resin falls from hopper (1) into vertical, cylindrical, high-intensity mixer (2) where additives also are placed. Hot dry-blend is fed into a cooling mixer (3), from which the cooled powder exits to another hopper (4) for fabrication

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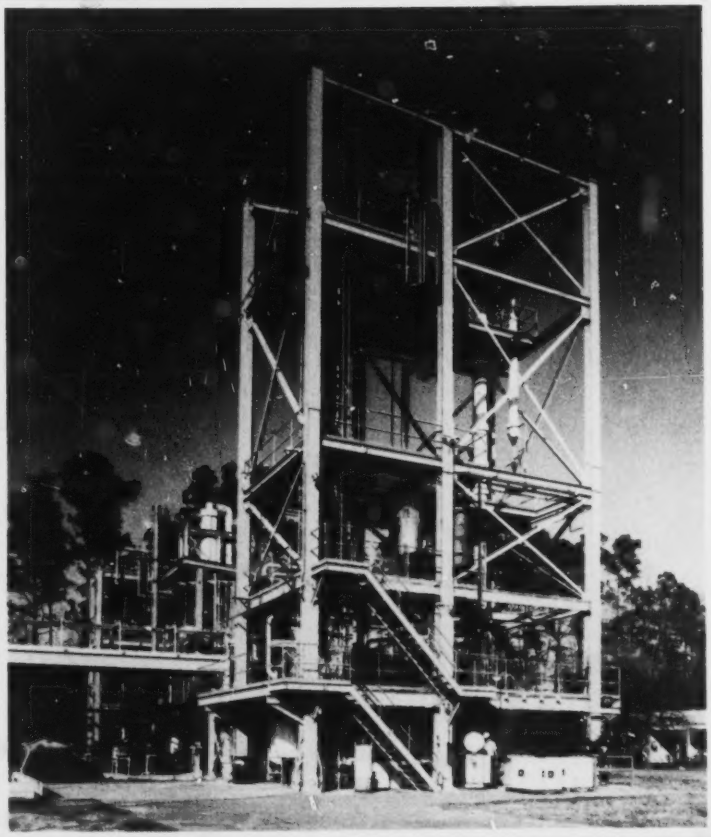
plasticizers and other additives is a viscous liquid that can be processed by techniques like spraying, dipping, knife or roll coating, and rotational and slush molding.

According to Cross Gates Consultants' Weinberg, PVC fabricators often prefer pelletized compound. This is because pellets are versatile, suitable for short runs. They are easier to store for long periods, to convey in materials handling equipment, to blend with any reground scrap or trim, and to fabricate with inexpensive, less sophisticated, conventional equipment. And pellets can be calendered, whereas powder cannot. In addition, though there are custom compounders that will supply powder, most fabricators who buy compound outside will buy pellets, and the supplier's quality control will make that of the buyer simpler. A disadvantage of pellets is that they are melt compounded, which adds more total exposure to degrading heat.

But above a certain use level, Weinberg says, it can be less costly to dry-blend powder in-house than to buy pellets outside. Dry blending subjects the compound to less total heat exposure and the mixers use less energy than the melt mixers needed for pellets. Also, with enough trial-and-error compounding as well as use of multiscrew injection molding machines or extruders to fabricate, compounders can get the same quality product at the same output rates as with pellets. Multiscrew machines use large amounts of shear and less external heating to produce homogeneous melts under mild conditions.

Whether dry- or melt-compounded, high-intensity mixing or premixing is often best. This is because such mixers volatilize excess vinyl chloride from the compound. Many high-intensity mixers are available for dry-blending PVC. One popular type is that of Littleford Brothers, Florence, Ky. A first mixer uses shear energy of blades to raise compound temperatures to 95 to 115 °C for complete absorption of plasticizer by PVC granules. This discharges into a cooling mixer, which rapidly drops compound temperature to 50 °C to densify the powder, render it more free-flowing, and avoid excessive exposure to heat.

Blades in the vertical, cylindrical, high-intensity mixer generate a vortex that moves the powder upward along the inner wall. At the top of the vortex, the blades throw the powder to the bottom, where it is again carried upward along the wall. In the horizontal, cylindrical cooling mixer, heat is removed by water circulating in the jacket, which is long and narrow to maximize contact surface. High-intensity mixing takes four to 10 minutes, depending on batch size. Cooling takes slightly less time, matched to exceed batch output rates of high-intensity mixing. The cooling mixer discharges either into a hopper container for



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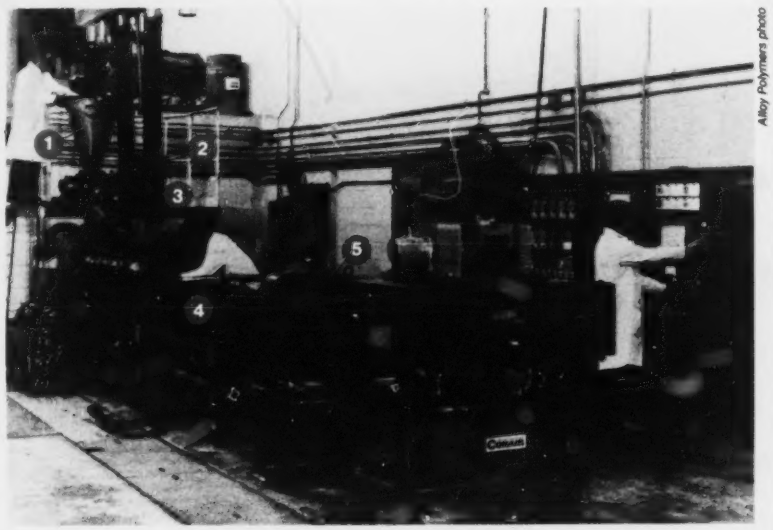

Resin already blended with some additives in a high-intensity mixer enters a twin-screw extruder (1) through a hopper. Additional fillers and colorants enter the extruder barrel (2). Then strands or sheet of compound exit the extruder through a die (3). A trough of water (4) cools the compound. Finally, a pelletizer or dicer (5) chops the compound strands or sheet

storage or to be taken to fabricating machines. Alternatively, cooled compound is transported overhead to production lines by an air-driven materials handling system.

Again, there are many systems for melt-compounding PVC. One popular type is a twin-screw compounding extruder of Werner & Pfleiderer Corp., Ramsey, N.J. Segmented construction of screws and barrels allows custom construction of the exact type needed. Multiscrew compounding adds more shear work to the compound, mixing it homogeneously without need for excessive external heating.

A second popular type is the single-screw kneader of Buss-Condux, Elk Grove Village, Ill. Screw flights are interrupted by gaps, and the extruder barrel is studded with teeth to match the gaps. As the screw turns, it also moves back and forth, producing both a mixing by shear against screw flight and barrel surfaces and a kneading by the teeth in the screw flight gaps. Such a machine operates at lower temperatures than ordinary single-screw machines because of lower internal pressures developed and because of controlled heating or cooling by circulating oil in the barrel and screw.

Product from the compounding extruder is pelletized or diced. Spaghettilike strands from the many holes in the extruder die are chopped into pellets, which drop into a container where they are fluidized and cooled by air. Alternatively, an extruded ribbon of compound passes through a long trough of cooling water, followed by dicing at the end.

High-speed mixers serve to disperse PVC in plasticizer to make plastisols. An example is that of Day Mixing, Cincinnati. A knife-edge rotor in the bottom of the vertical cylinder spins at 9000 rpm and sets up a vortex of liquid, which impinges against baffles set in the inner wall. In addition to the rotary motion, there is convection of liquid up the inner walls of the mixer and down the interior of the vortex to contact the rotor. Cooling water at 15 °C in an outer jacket keeps plastisol temperatures safely below 35 °C. At the end of a 15-minute mixing cycle, a vacuum of five minutes' duration deaerates the plastisol. □

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